

PCT

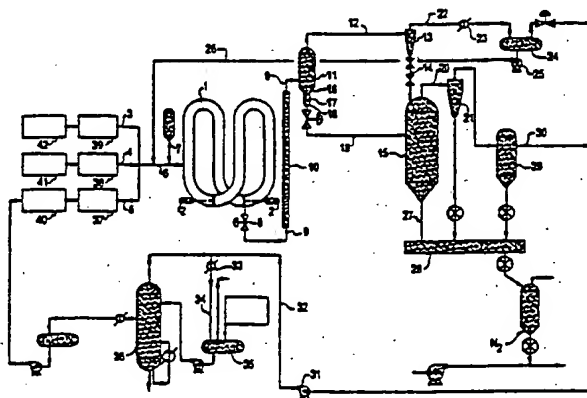
WORLD INTELLECTUAL PROPERTY ORGANIZATION  
International Bureau



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification <sup>6</sup> : B01J 8/00, B01D 3/06, B01J 19/18, C08F 6/00		A1	(11) International Publication Number: WO 99/47251
		(43) International Publication Date: 23 September 1999 (23.09.99)	
(21) International Application Number: PCT/US99/06102		(81) Designated States: AU, BR, CA, CN, IN, JP, KR, MX, RU, SG, ZA, Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).	
(22) International Filing Date: 19 March 1999 (19.03.99)			
(30) Priority Data: 60/078,859 20 March 1998 (20.03.98) US 09/080,412 18 May 1998 (18.05.98) US 09/081,392 18 May 1998 (18.05.98) US		Published With international search report. Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.	
(71) Applicant: EXXON CHEMICAL PATENTS INC. [US/US]; 5200 Bayway Drive, Baytown, TX 77520-5200 (US).			
(72) Inventor: KENDRICK, James, Austin, 10314 Sagefield Drive, Baton Rouge, LA 70811 (US).			
(74) Agent: REIDY, Joseph, F.; Exxon Chemical Company, P.O. Box 2149, Baytown, TX 77522-2149 (US).			

(54) Title: CONTINUOUS SLURRY POLYMERIZATION VOLATILE REMOVAL



(57) Abstract

A process/apparatus is disclosed for continuously separating a liquid medium comprising diluent and unreacted monomers from a polymerization effluent comprising diluent, unreacted monomers and polymer solids, comprising a continuous discharge of the polymerization effluent from a slurry reactor through a discharge valve and transfer conduit into a first intermediate pressure flash tank with a conical bottom defined by substantially straight sides inclined at an angle to that of horizontal equal to or greater than the angle of slide of the slurry/polymer solids and an exit seal chamber of such diameter (d) and length (l) as to maintain a desired volume of concentrated polymer solids/slurry in the exit seal chamber such as to form a pressure seal while continuously discharging a plug flow of concentrated polymer solids/slurry bottom product of said first flash tank from the exit seal chamber through a seal chamber exit reducer with inclined sides defined by substantially straight sides inclined at an angle to that of horizontal equal to or greater than the angle of slide of the polymer solids which remain after removal of about 50 to 100 % of the inert diluent therefrom to a second flash tank at a lower pressure.

*FOR THE PURPOSES OF INFORMATION ONLY*

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AL	Albania	ES	Spain	LS	Lesotho	SI	Slovenia
AM	Armenia	FI	Finland	LT	Lithuania	SK	Slovakia
AT	Austria	FR	France	LU	Luxembourg	SN	Senegal
AU	Australia	GA	Gabon	LV	Latvia	SZ	Switzerland
AZ	Azerbaijan	GB	United Kingdom	MC	Monaco	TD	Chad
BA	Bosnia and Herzegovina	GE	Georgia	MD	Republic of Moldova	TG	Togo
BB	Barbados	GH	Ghana	MG	Madagascar	TJ	Tajikistan
BE	Belgium	GN	Guinea	MK	The former Yugoslav Republic of Macedonia	TM	Turkmenistan
BF	Burkina Faso	GR	Greece	ML	Mali	TR	Turkey
BG	Bulgaria	HU	Hungary	MN	Mongolia	TT	Trinidad and Tobago
BJ	Benin	IE	Ireland	MR	Mauritania	UA	Ukraine
BR	Brazil	IL	Israel	MW	Malawi	UG	Uganda
BY	Belarus	IS	Iceland	MX	Mexico	US	United States of America
CA	Canada	IT	Italy	NE	Niger	UZ	Uzbekistan
CF	Central African Republic	JP	Japan	NL	Netherlands	VN	Viet Nam
CG	Congo	KE	Kenya	NO	Norway	YU	Yugoslavia
CH	Switzerland	KG	Kyrgyzstan	NZ	New Zealand	ZW	Zimbabwe
CI	Côte d'Ivoire	KP	Democratic People's Republic of Korea	PL	Poland		
CM	Cameroon	KR	Republic of Korea	PT	Portugal		
CN	China	KZ	Kazakhstan	RO	Romania		
CU	Cuba	LC	Saint Lucia	RU	Russian Federation		
CZ	Czech Republic	LJ	Liechtenstein	SD	Sudan		
DE	Germany	LK	Sri Lanka	SE	Sweden		
DK	Denmark	LR	Liberia	SG	Singapore		
EE	Estonia						

**CONTINUOUS SLURRY  
POLYMERIZATION VOLATILE REMOVAL**

**FIELD OF INVENTION**

5           The present invention relates to an apparatus for continuously separating polymer solids from a liquid medium comprising an inert diluent and unreacted monomers. In particular, the present invention relates to an apparatus for continuously separating polymer solids from a liquid medium, drying the polymer, and recovering the diluent and unreacted monomers with a reduction in  
10       compression needed for diluent vapor condensation to liquid diluent for reuse in a polymerization process. In another aspect, the invention relates to a method for continuously separating polymer solids from a liquid medium. In particular, the invention relates to a method for continuously separating polymer solids from a liquid medium, drying the polymer, and recovering the inert diluent and unreacted  
15       monomers for reuse in a polymerization process.

**BACKGROUND OF THE INVENTION**

          In many polymerization processes for the production of polymer, a polymerization effluent is formed which is a slurry of particulate polymer solids  
20       suspended in a liquid medium, ordinarily the reaction diluent and unreacted monomers. A typical example of such processes is disclosed in Hogan and Bank's U.S. Pat. No. 2,285,721, the disclosure of which is incorporated herein by reference. While the polymerization processes described in the Hogan document employs a catalyst comprising chromium oxide and a support, the present  
25       invention is applicable to any process producing an effluent comprising a slurry of particulate polymer solids suspended in a liquid medium comprising a diluent and unreacted monomer. Such reaction processes include those which have come to be known in the art as particle form polymerizations.

          In most commercial scale operations, it is desirable to separate the polymer  
30       and the liquid medium comprising an inert diluent and unreacted monomers in such a manner that the liquid medium is not exposed to contamination so that the liquid medium can be recycled to the polymerization zone with minimal if any

-2-

purification. A particularly favored technique that has been used heretofore is that disclosed in the Scoggin et al, U.S. Pat. No. 3,152,872, more particularly the embodiment illustrated in conjunction with Fig. 2 of that patent. In such processes the reaction diluent, dissolved monomers, and catalyst are circulated in a loop reactor wherein the pressure of the polymerization reaction is about 100 to 700 psia. The produced solid polymer is also circulated in the reactor. A slurry of polymer and the liquid medium is collected in one or more settling legs of the slurry loop reactor from which the slurry is periodically discharged to a flash chamber wherein the mixture is flashed to a low pressure such as about 20 psia. While the flashing results in substantially complete removal of the liquid medium from the polymer, it is necessary to recompress the vaporized polymerization diluent (i.e., isobutane) in order to condense the recovered diluent to a liquid form suitable for recycling as liquid diluent to the polymerization zone. The cost of compression equipment and the utilities required for its operation often amounts to a significant portion of the expense involved in producing polymer.

Some polymerization processes distill the liquefied diluent prior to recycling to the reactor. The purpose of distillation is removal of monomers and light-end contaminants. The distilled liquid diluent is then passed through a treater bed to remove catalyst poisons and then on to the reactor. The equipment and utilities costs for distillation and treatment can be a significant portion of the cost of producing the polymer.

In a commercial scale operation, it is desirable to liquefy the diluent vapors at minimum cost. One such technique used heretofore is disclosed in Hanson and Sherk's U.S. Pat. No. 4,424,341 in which an intermediate pressure flash step removes a significant portion of the diluent at such a temperature and at such a pressure that this flashed portion of diluent may be liquified by heat exchange instead of by a more costly compression procedure.

### BRIEF SUMMARY OF THE INVENTION

The present invention relates to an apparatus for continuously separating polymer solids from a liquid medium comprising an inert diluent and unreacted monomers. In another aspect, the invention relates to an apparatus for continuously separating polymer solids from a liquid medium, drying the polymer, and recovering the diluent and unreacted monomers with a reduction in compression needed for diluent vapor condensation to liquid diluent for reuse in a polymerization process. In another aspect, the invention relates to a method for continuously separating polymer solids from a liquid medium. In another aspect, the invention relates to a method for continuously separating polymer solids from a liquid medium, drying the polymer, and recovering the inert diluent and unreacted monomers for reuse in a polymerization process.

In accordance with the present invention, there is provided an apparatus for continuously recovering polymer solids from a polymerization effluent comprising a slurry of said polymer solids in a liquid medium comprising an inert diluent and unreacted monomers. The apparatus comprises a discharge valve on a slurry reactor, examples of which include slurry loop reactors and stirred tank slurry reactors, for the continuous discharge of a portion of the slurry reactor contents into a first transfer conduit: a first flash tank having a bottom defined by substantially straight sides inclined at an angle to the horizontal equal to or greater than the angle of slide of the slurry/polymer solids; wherein the pressure of the first flash tank and temperature of the polymerization effluent are such that from about 50% to about 100% of the liquid medium will be vaporized and the inert diluent component of said vapor is condensable, without compression, by heat exchange with a fluid having a temperature in the range of about 65°F to about 135°F; a first flash tank exit seal chamber, communicating with said first flash tank, of such a length (l) and diameter (d) as to permit such a level of concentrated polymer solids/slurry to accumulate and form a pressure seal in said first flash tank exit seal chamber; a seal chamber exit reducer providing for a continuous discharge of a plug flow of concentrated polymer solids/slurry to a second transfer conduit which communicates the concentrated polymer solids/slurry into a second

flash tank wherein the pressure of said second flash tank and temperature of the concentrated polymer solids/slurry are such that essentially all of any remaining inert diluent and/or unreacted monomer will be vaporized and removed overhead for condensation by compression and heat exchange and the polymer solids are  
5 discharged from the bottom of said second flash tank for additional processing or storage.

The invention provides also a method for the continuous removal of a stream of polymerization effluent from a slurry reactor through a discharge valve; increasing the heat content of the polymerization effluent during its transit through  
10 said first transfer conduit to a temperature below the fusion point of the polymer while continuously communicating the polymerization effluent to a first flash tank having a bottom defined by substantially straight sides inclined at an angle to the horizontal equal to or greater than the angle of slide of the concentrated polymer solids/slurry; continuously vaporizing from about 50% to about 100% of the  
15 liquid medium in said first heated flash tank to yield a concentrated polymer solids/slurry and a vapor stream at such a temperature and pressure that the inert diluent content of said vapor is condensable, without compression, by heat exchange with a fluid having a temperature in the range from about 65° F to about 135° F; continuously discharging the concentrated polymer solids/slurry from said  
20 first flash tank to a first flash tank exit seal chamber of such a length (l) and diameter (d) that a volume of concentrated polymer solids/slurry is continuously maintained so as to form a pressure seal in said first flash tank exit seal chamber; continuously discharging the concentrated polymer solids/slurry from said first flash tank seal chamber through a seal chamber exit reducer defined by  
25 substantially straight sides inclined at an angle to that of horizontal equal to or greater than the angle of slide of the polymer solids which remain after removal of about 50 to 100% of the inert diluent therefrom; communicating a continuous plug flow of concentrated polymer solids/slurry from said first flash tank exit seal chamber through said seal chamber exit reducer to a second transfer conduit  
30 which communicates said continuous plug flow of concentrated polymer solids/slurry to a second flash tank; and continuously vaporizing essentially all of any remaining inert diluent and/or unreacted monomer in a second flash tank

operated at a lower pressure than said first flash tank; condensing the vaporized inert diluent and/or unreacted monomer from said second flash tank by compression and heat exchange; and continuously discharging the essentially dried polymer slurry from said second flash tank for further processing or storage.

5       An object of the present invention is to provide both an apparatus and method for the continuous two stage flash drying of the polymer solids following the continuous removal of the polymerization effluent comprising polymer solids and liquid medium comprising inert diluent and unreacted monomers from a slurry reactor through a point discharge valve, a continuous solids level control in  
10   the first flash tank exit seal chamber that provides a pressure seal therein which enables said first flash tank to operate under a substantially greater pressure than said second flash tank while polymer solids are continuously discharged through the seal chamber exit reducer into the second transfer conduit and further into the second flash tank which eliminates plugging in the first flash tank and the  
15   continuous liquification of from about 50% to about 100% of the inert diluent vapor by heat exchange rather than compression.

Another object of the invention is to eliminate the need for a settling leg on the slurry reactor and the intermittent high pressure pulse in the slurry reactor caused by periodic discharging of the contents of the settling leg. Another object  
20   of the present invention is to improve safety by eliminating the possibility of plugging in a settling leg.

Another object of the invention is to eliminate plugging in equipment downstream from the discharge valve. In a settling leg of a polymerization reactor polymerization continues and the heat of reaction further heats the liquid medium  
25   and a potential exists for some of the polymer solids to dissolve or to fuse together. As the contents of the settling leg exit the discharge valve, the pressure drop causes flashing of some of the liquid medium which results in cooling the remaining liquid medium causing the dissolved polymer to precipitate which tends to plug downstream equipment. The present invention which eliminates the need  
30   for a settling leg also eliminates this potential for downstream equipment plugging by avoiding the initial dissolution or fusion of the polymer solids.

Another object of the present invention is to increase the reactor throughput by the use of increased ethylene concentrations in the liquid medium, e.g., greater than or equal to 4 weight percent at reactor outlet. Settling legs limit ethylene concentrations due to an increased tendency to plug downstream equipment caused by accelerated reaction within the settling leg. A continuous polymerization effluent slurry flow allows ethylene concentrations to be limited only by the ethylene solubility in the liquid diluent in the reactor, thereby increasing the specific reaction rate for polymerization and increasing reactor throughput.

Other aspects, objects, and advantages of the present invention will be apparent from the following disclosure and Figures 1 and 2.

The claimed apparatus and process provide several advantages over the prior art including: (1) allowing for a continuous processing of the contents of a slurry reactor from the point of discharge of the liquified polymerization effluent through a discharge valve; a first flash tank; a seal chamber; a seal chamber exit reducer; and therefrom to a second flash tank, (2) significantly increasing ethylene concentration in the liquid medium thereby increasing reactor through-put and (3) energy consumption is reduced by reducing the need to compress and/or distill the reactor vapor-liquid effluent. Recycling compressors and other downstream equipment can be reduced in size or eliminated.

#### **BRIEF DESCRIPTION OF THE DRAWING**

Figs. 1 and 2 are a schematic diagram illustrating an apparatus for continuously separating polymer solids from diluent and unreacted monomer in accordance with the present invention.

#### **DETAILED DESCRIPTION OF THE INVENTION**

The present invention is applicable to any mixture which comprises a slurry of polymer solids and a liquid medium comprising an inert diluent and unreacted monomers including slurries resulting from olefin polymerization. The olefin monomers generally employed in such reactions are 1-olefins having from



2 up to 8 carbon atoms per molecule. Typical examples include ethylene, propylene, butene, pentene, hexene and octene.

Typical diluents employed in such olefin polymerizations include saturated aliphatic hydrocarbons having 3 to 8, preferably 3 to 4 carbon atoms per molecule, such as propane, isobutane, propylene, n-butane, n-pentane, isopentane, n-hexane, isooctane, and the like. Of these diluents those of 3 to 4 carbon atoms per molecule are preferred, and isobutane is most preferred.

The rate of discharge of the polymerization effluent is such as to allow a continuous process stream from the slurry loop reactor from the point of discharge of the liquified polymerization effluent through a single point discharge valve and also through the first flash tank and the associated vapor recovery and solids recovery systems. The rate of discharge of the polymerization effluent is such as to maintain a constant pressure in the slurry reactor and to eliminate intermittent high pressure pulses associated with a discharge of a portion of the reactor contents that occurs with settling legs on slurry reactors.

The temperature to which the polymerization effluent slurry which is discharged from the reactor is heated during transit to the first flash tank for vaporization is below the fusion temperature of the polymer. This may be accomplished by appropriate heating of this first transfer conduit. The quantity of heat to be supplied to the polymerization effluent during its transit through this first conduit to the first flash tank should preferably be at least equal to that quantity of heat which equals the heat of vaporization of that quantity of inert diluent which is to be flash vaporized in the first flash tank. This then will provide for the concentrated polymer solids formed in the first flash tank to be passed to the second flash tank to pass thereto at a higher solids temperature and thus facilitates the removal of residual diluent in the pores of such polymer solids by the operation of the second flash tank. That quantity of heat transferred to the polymerization effluent during its transit through the first transfer conduit to the first flash tank may even be greater, provided only that the quantity of heat so transferred will not cause the polymer solids therein to become heated to such a temperature at which they will tend to fuse or agglomerate one with another.

The concentrated polymer solids/slurry are discharged from the first flash tank into a first flash tank exit seal chamber of such a length (l) and diameter (d) so as to provide a volume sufficient to maintain a volume of concentrated polymer solids/slurry sufficient to maintain a pressure seal in the exit seal chamber. The concentrated polymer solids/slurry are discharged from the exit seal chamber through an exit seal chamber reducer to a second transfer conduit which communicates the concentrated polymer solids/slurry as a plug flow to a second flash tank. The exit seal chamber reducer is defined by substantially straight sides inclined at an angle to that of horizontal equal to or greater than the angle of slide of the concentrated polymer solids/slurry.

The pressure for the first flash step will vary depending on the nature of the diluent and unreacted monomers and the temperature of the polymerization effluent. Typically, pressures in the range of from about 140 psia to about 315 psia can be employed; more preferably from about 200 psia to about 270 psia; and most preferably from about 225 psia to about 250 psia.

The heat exchanging fluid used to condense the vapor from the first flash step is at a temperature in the range of from about 65°F to about 135°F. A preferred embodiment uses a heat exchange fluid at a temperature of from about 75°F to about 125°F. A most preferred embodiment uses a heat exchange fluid at a temperature of from about 85°F to about 115°F.

A further understanding of the present invention will be provided by referring to Fig. 1 which illustrates a system comprising an embodiment of the invention.

In the embodiment illustrated in Fig. 1, the polymerization is carried out in a loop reactor 1. The polymerization mixture is circulated by agitator 2. Diluent comonomer and monomer are introduced from the diluent storage vessel 40, the comonomer storage vessel 41, and the monomer storage vessel 42 through their respective treater beds 37, 38, and 39 through conduits 5, 4 and 3, respectively, connected to conduit 6. Catalyst is added through conduit 7. Normally, catalyst is introduced as a suspension in a hydrocarbon diluent.

Polymerization effluent is removed from the loop by continuous discharge through the single point discharge valve 8. The polymerization effluent passes

from the discharge valve 8 to a conduit 9 which is provided with a line heater 10 and into the first flash tank 11 which separates vaporized liquid medium from polymer slurry/solids. Conduit 9 has an indirect heat exchange means such as a flash line heater 10.

5 Vaporized liquid medium comprising diluent and unreacted monomers exit the first flash tank 11 via transfer conduit 12 through which it is passed into a cyclone 13 which separates entrained polymer solids from the vapor. Polymer solids separated by the cyclone are passed via line 14 through a dual valving assembly designed to maintain a pressure seal below cyclone 13 to a lower  
10 pressure flash tank 15.

The concentrated polymer solids/slurry in the bottom of the first flash tank 11 continuously settles by sliding along the straight line bottom surface 16 thereof into the seal chamber 17 which is illustrated in enlargement Figure 2. A polymer solids/slurry level 43 is maintained in the seal chamber 17 to eliminate plugging  
15 tendencies in flash tank 11 and to form a pressure seal so that flash tank 11 can operate at a substantially higher pressure than flash tank 15. Polymer slurry/solids are continuously discharged from the seal chamber 17 into the lower pressure flash tank 15. The length (l), diameter (d), and volume of the seal chamber 17 and the geometry of the seal chamber exit reducer 18 are chosen so as to provide a  
20 variable residence time and provide a continuous plug flow of concentrated polymer solids/slurry to minimize "dead" space and reduce plugging tendencies. The seal chamber 17 length must be sufficient to allow practical level measurement and control. Typical residence times of the concentrated polymer solid/slurry in the seal chamber 17 are from 5 seconds to 10 minutes, preferable  
25 residence times are from 10 seconds to 2 minutes and most preferable residence times from 15 - 45 seconds. The continuous plug flow of concentrated polymer solids/slurry forms a pressure seal wherein the concentrated polymer solids/slurry have an  $l/d$  ratio inside the seal chamber 17 which is typically 1.5 to 8, preferable  $l/d$  is 2 to 6 and most preferable is 2.2 to 3. Typically the seal chamber exit  
30 reducer 18 sides are inclined, relative to the horizontal, 60 - 85 degrees, preferable 65 - 80 degrees and most preferable 68 - 75 degrees. The seal chamber exit reducer 18 geometry is defined by substantially straight sides inclined at an angle

-10-

to that of horizontal equal to or greater than the angle of slide of the concentrated polymer slurry/solids and communicates the concentrated polymer solid/slurry to a second transfer conduit 19 which communicates with a feed inlet of flash tank 15. In flash tank 15 substantially all of any remaining inert diluent and unreacted monomer in the concentrated polymerization effluent is vaporized and taken  
5 overhead via conduit 20 to a second cyclone 21.

The major portion of the liquid medium in the polymerization effluent will have been taken to cyclone 13 as vapor. The vapor after having the entrained polymer solids removed is passed via conduit 22 through a heat exchanger 23  
10 wherein the vapor at a pressure from about 140 psia to about 315 psia is condensed by indirect heat exchange with a heat exchange fluid such as to eliminate the need for compression.

The condensed liquid medium comprising diluent and unreacted monomers is then passed to an accumulator 24. A pump 25 is provided for  
15 conveying the condensed liquid medium back to the polymerization zone by line 26.

The polymer solids in the lower pressure flash tank 15 are passed via line 27 to a conventional dryer 28. The vapor exiting the secondary cyclone 21, after filtration in filter unit 29, is passed by line 30 to a compressor 31 and the  
20 compressed vapors are passed through a conduit 32 to a condenser 33 where vapor is condensed and the condensate is passed through conduit 34 to storage vessel 35. The condensed liquid medium in the storage vessel 35 is typically vented overhead for removal of light-end contaminants. The inert diluent can be returned to the process through a treater bed 37 to remove catalyst poisons or distilled in  
25 unit 36 for more complete removal of light-ends and then returned to the process through a treater bed.

Having broadly described the present invention it is believed that the same will become even more apparent by reference to the following examples. It will be appreciated that the examples are presented solely for the purpose of  
30 illustration and should not be construed as limiting the invention.

### EXAMPLES

#### Example 1

A typical ethylene polymerization process would be conducted at a temperature of about 215°F and a pressure of 565 psia. An example of such a process would result in a polymerization effluent of about 83,000 pounds per hour comprising about 45,000 pounds per hour of polyethylene polymer solids and about 38,000 pounds per hour of isobutane and unreacted monomers. The continuously discharged polymerization effluent is flashed in the first flash tank at a pressure of about 240 psia and a temperature of about 180°F to remove overhead about 35,000 pounds per hour of diluent and unreacted monomer vapors and entrained particulates. Auxiliary heat to impart an additional quantity of heat to the polymerization effluent is supplied by appropriate heating means during the transit between the discharge valve and the first flash tank. After removal of the fines, the isobutane vapor is condensed, without compression, by heat exchange at a pressure of about 240 psia and a temperature of about 135°F. The polymer slurry/solids discharging from the bottom of the first flash tank into the seal chamber form a continuous plug flow of concentrated polymer slurry/solids, which provides a pressure seal, with an L/d ratio of the plug of polymer slurry/solids of 2.5 in an 8'4" long seal chamber having an L/d ratio of 5.5 and with a cone angle of about 68° on the seal chamber exit reducer. The residence time of the continuous plug flow of concentrated polymer slurry/solids is about 16 seconds. The concentrated polymer slurry/solids are continuously discharged from the bottom of the first flash tank at a temperature of about 180°F and a pressure of about 240 psia through a seal chamber, seal chamber exit reducer, and a second transfer conduit into a feed inlet on a second flash tank. The remaining liquid medium in the concentrated polymer slurry/solids communicated to the second flash tank is flashed at a temperature of about 175°F and at a pressure of about 25 psia to remove about 4,300 pounds per hour of isobutane and unreacted monomers which are condensed by compression and heat exchange.

#### Example 2

A typical ethylene polymerization process would be conducted at a temperature of about 215°F and a pressure of 565 psia. An example of such a

-12-

process would result in a polymerization effluent of about 83,000 pounds per hour comprising about 45,000 pounds per hour of polyethylene polymer solids and about 38,000 pounds per hour of isobutane and unreacted monomers. The continuously discharged polymerization effluent is flashed in the first flash tank at a pressure of about 240 psia and a temperature of about 175° F to remove overhead about 23,000 pounds per hour of diluent and unreacted monomer vapors and entrained particulates. After removal of the fines, the isobutane vapor is condensed, without compression, by heat exchange at a pressure of about 240 psia and a temperature of about 112° F. The polymer slurry/solids discharging from the bottom of the first flash tank into the seal chamber form a continuous plug flow of concentrated polymer slurry/solids, which provides a pressure seal, with an l/d ratio of the plug of polymer slurry/solids of 2.5 in an 8'4" long seal chamber with an l/d ratio of 5.5 and with a cone angle of about 68° on the seal chamber exit reducer. The residence time of the continuous plug flow of concentrated polymer slurry/solids in the seal chamber is about 16 seconds. About 60,000 pounds per hour of concentrated polymer slurry/solids are continuously discharged from the bottom of the first flash tank at a temperature of about 175° F and a pressure of about 240 psia through a seal chamber, seal chamber exit reducer and a second transfer conduit into a feed inlet on a second flash tank. The remaining liquid medium in the concentrated polymer slurry/solids communicated to the second flash tank is flashed at a temperature of about 125° F and at a pressure of about 25 psia to remove about 16,000 pounds per hour of isobutane and unreacted monomer which are condensed by compression and heat exchange.

CLAIMS

What is claimed is:

1. An apparatus for removing and recovering polymerization liquid  
5 medium from a polymer produced in a reactor as a polymer slurry of particulate  
polymer solids suspended in a liquid medium comprising an inert diluent and  
unreacted monomers, the apparatus comprising,  
a discharge valve for continuously discharging a portion of said polymer  
slurry from said reactor into a first transfer conduit;  
10 said first transfer conduit communicating said polymer slurry therein into a  
first flash tank having a bottom communicating to a first flash tank exit seal  
chamber of a length (l) and a diameter (d) which provides a volume sufficient to  
maintain a volume of polymer solids/slurry sufficient to maintain a pressure seal  
in said seal chamber;  
15 said seal chamber having a seal chamber exit reducer, defined by  
substantially straight sides inclined at an angle to that of horizontal equal to or  
greater than the angle of slide of the polymer solids which remain after removal of  
about 50 to 100% of the inert diluent therefrom which communicates a continuous  
flow of concentrated polymer solids/slurry from said first flash tanks exit seal  
20 chamber to a second transfer conduit which communicates said continuous flow  
of concentrated polymer solids/slurry to a second flash tank; and  
said second flash tank operating at a substantially lesser pressure than that  
of said first flash tank such that essentially all of any remaining inert diluent and  
monomer is vaporized and communicated to a diluent and monomers recovery  
25 system through a flash tank overhead exit and essentially dried polymer solids are  
communicated to a dryer/storage tank.
2. The apparatus of claim 1 wherein the first flash tank has a bottom  
defined by substantially straight sides inclined at an angle to that of horizontal  
equal to or greater than the angle of slide of the concentrated polymer solids/slurry  
30 which remain after removal of about 50 to 100% of the inert diluent therefrom.
3. An apparatus of claim 1 wherein said first flash tank exit seal  
chamber has a volume of a continuous plug flow of said concentrated polymer

wherein said volume of continuous plug flow of said concentrated polymer/slurry solids has a length (l) to diameter (d) ratio (l/d) of from about 1.5 to about 8.0.

4. An apparatus of claim 3 wherein said first transfer conduit is heated by a heater means capable of providing a quantity of heat sufficient to  
5 maintain said polymer slurry therein at a temperature below the fusion temperature of the polymer

5. A process for producing polymer comprising producing a polymer slurry in a liquid medium which comprises:

reacting a monomer in a hydrocarbon diluent inert to polymerization to  
10 form a polymerization effluent;  
continuously discharging said polymerization effluent through a discharge valve into a first transfer conduit;

heating said polymerization effluent in said first transfer conduit to a temperature below the fusion temperature of the polymer;

15 continuously communicating said polymerization effluent through said first transfer conduit to a first flash tank wherein the pressure in said first flash tank and the temperature of said heated polymerization effluent are such as to produce as a vapor from about 50% to about 100% of the liquid medium and said vapor is condensable, without compression, by heat exchange with a fluid having  
20 a temperature in the range of about 65° F to about 135° F;

continuously condensing said vapor obtained in said first flash step, without compression, by heat exchange with a fluid having a temperature in the range of about 65° F to about 135° F;

continuously discharging from said first flash tank concentrated polymer  
25 solids/slurry to a second flash tank through a seal chamber wherein said seal chamber has a length (l) and a diameter (d) such as to maintain a volume of concentrated polymer solids/slurry in the said seal chamber sufficient to maintain a pressure seal;

continuously communicating said concentrated polymer solids/slurry to a  
30 second flash tank through a seal chamber exit reducer defined by substantially straight sides inclined at an angle to that of horizontal equal to or greater than the



angle of slide of the polymer solids which remain after removal of about 50 to 100% of the inert diluent therefrom;

continuously exposing the remaining liquid medium in said concentrated polymer solids/slurry to a further pressure reduction from a higher pressure of  
5 from about 140 psia to about 315 psia in said first flash tank to a lower pressure of from about 15 psia to about 35 psia in said second flash tank wherein the pressure of said second flash tank and the temperature of said heated concentrated polymer slurry are such as to produce as a vapor substantially all of the remaining diluent and monomer and said vapor is condensable with compression and cooling; and  
10 discharging from said second flash tank polymer solids which are substantially free of diluent or unreacted monomer.

6. The process of claim 5 wherein the first flash tank has a bottom defined by substantially straight sides inclined at an angle to that of horizontal equal to or greater than the angle of slide of the concentrated polymer solids/slurry  
15 which remain after removal of about 50 to 100% of the inert diluent therefrom.

7. The process of claim 5 wherein said first transfer conduit is heated by a heater means of a heating capacity capable of providing a quantity of heat sufficient to bring said polymer slurry therein to a temperature below the fusion temperature of the polymer solids.

20 8. The process of claim 7 wherein said liquid diluent is isobutane.

9. The process of claim 5 wherein the liquid medium comprises increased ethylene concentrations greater than or equal to 4 weight percent at the reactor outlet.

10. The process of claim 5 wherein the rate of discharge of the polymerization effluent maintains a constant pressure in the slurry reactor.

11. The process of claim 5 wherein the residence time of the concentrated polymer solid/slurry in the seal chamber are from 5 seconds to 10 minutes.

12. The process of claim 5 wherein the residence time of the concentrated polymer solid/slurry in the seal chamber are from 10 seconds to 2  
30 minutes.

13. The process of claim 5 wherein the residence time of the concentrated polymer solid/slurry in the seal chamber are from 15 seconds to 45 seconds.

14. The process of claim 5 wherein substantially any remaining inert  
5 diluent and unreacted monomer in the concentrated polymerization effluent in the second flash tank is vaporized and removed.

15. The apparatus of claim 1 wherein the discharge valve discharges the polymerization effluent at a rate to maintain a constant pressure in the slurry reactor.

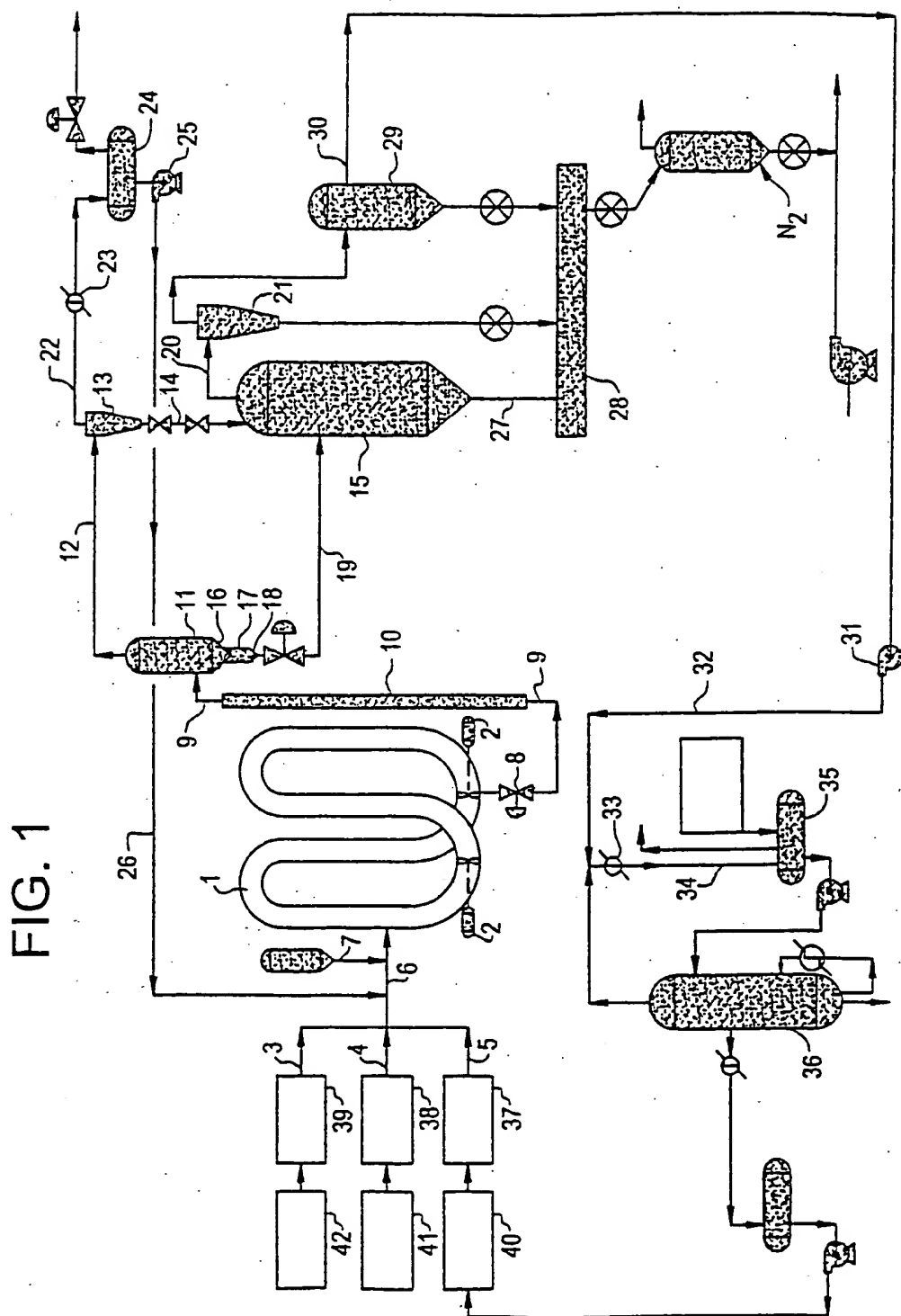
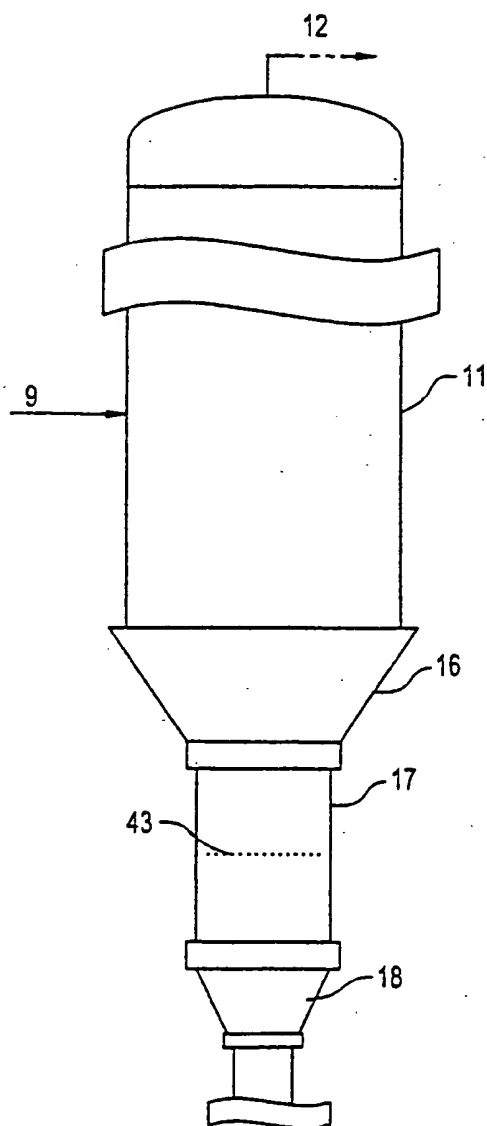


FIG. 1

**SUBSTITUTE SHEET (RULE 26).**

FIG. 2



# INTERNATIONAL SEARCH REPORT

International Application No  
PCT/US 99/06102

A. CLASSIFICATION OF SUBJECT MATTER  
IPC 6 B01J8/00 B01D3/06 B01J19/18 C08F6/00

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 C08F

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 4 424 341 A (HANSON DONALD O ET AL) 3 January 1984 (1984-01-03) cited in the application abstract; claims; figure; example ---	1-15
X	US 5 575 979 A (HANSON DONALD O) 19 November 1996 (1996-11-19) abstract; claims; figures ---	1-15
X	EP 0 415 427 A (PHILLIPS PETROLEUM CO) 6 March 1991 (1991-03-06) abstract; claims; figure -----	1-15

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

### \* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"Z" document member of the same patent family

Date of the actual completion of the international search

22 July 1999

Date of making of the international search report

30/07/1999

Name and mailing address of the ISA

European Patent Office, P.B. 5816 Patentaan 2  
NL - 2280 HV Rijswijk  
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,  
Fax: (+31-70) 340-3016

Authorized officer

Mettler, R-M

# INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No.

PCT/US 99/06102

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 4424341 A	03-01-1984	NONE	
US 5575979 A	19-11-1996	US 5597892 A	28-01-1997
EP 0415427 A	06-03-1991	US 5183866 A	02-02-1993
		AT 122689 T	15-06-1995
		CA 2018920 A,C	28-02-1991
		DE 69019439 D	22-06-1995
		DE 69019439 T	14-09-1995
		ES 2072340 T	16-07-1995
		HU 209740 B	28-10-1994
		JP 2701968 B	21-01-1998
		JP 3093806 A	18-04-1991
		MX 171494 B	29-10-1993
		NO 175209 B	06-06-1994